Results of Proficiency Test Aviation gasoline April 2012

Organised by: Institute for Interlaboratory Studies Spijkenisse, the Netherlands

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### **1** INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise a new proficiency test for the analysis of Aviation Gasoline during the annual proficiency testing program 2011/2012. In this interlaboratory study 19 laboratories in 17 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the Aviation Gasoline proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

## 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. It was decided to evaluate the Aviation Gasoline according to the test scope of ASTM D910:11. The analyses for fit-for-use and homogeneity testing were subcontracted. In this proficiency test, the participants received one sample of Aviation Gasoline (Avgas 100LL).

Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

## 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO17043:2010 and ILAC-G13:2007. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Also customer's satisfaction is measured on a regular basis by sending out questionnaires.

## 2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2), which can be downloaded from www.iisnl.com.

### 2.3 CONFIDENTIALITY STATEMENT

All data present in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

## 2.4 SAMPLES

The necessary sample was obtained from an European supplier. After homogenisation, the material was transferred into 74 brown glass bottles of 1 litre (#12050). The homogeneity of the subsamples #12050 was checked by determination of Density @15°C in accordance with ASTM D4052:11 on 8 stratified randomly selected samples.

	Density @ 15°C in kg/m <sup>3</sup>
Sample #12050-1	710.77
Sample #12050-2	710.80
Sample #12050-3	710.79
Sample #12050-4	710.79
Sample #12050-5	710.78
Sample #12050-6	710.74
Sample #12050-7	710.78
Sample #12050-8	710.80

table 1: homogeneity test results of subsamples #12050

From the above test results, the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method which is in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @ 15 °C in kg/m <sup>3</sup>
r (sample #12050)	0.02
reference test	D4052:11
0.3*R (reference test)	0.97

table 2: evaluation of repeatability of the subsamples #12050

The calculated repeatability is less than 0.3 times the reproducibility of the corresponding reference method. Therefore, homogeneity of the subsamples #12050 was assumed.

To the participants one 1L bottle with sample #12050 was sent on 28 March, 2012.

## 2.5 ANALYSIS

The participants were requested to determine on sample #12050: Colour, Copper Strip Corrosion 2 hrs/100 °C, Density, Distillation, Existent Gum, Freezing Point, Heat of Combustion (Net), Lead as Pb, Lead as TEL, Lead participate, MON, Potential Gum, Sulphur and Water reaction interface (volume change).

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website.

A SDS and a form to confirm receipt of the samples were added to the sample package.

## 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

## 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

## 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nr.13 and 14).

## 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate the fit-for-useness of the reported test result.

The z-scores were calculated in accordance with:

 $z_{(target)} = (result - average of PT) / target standard deviation$ 

The  $z_{(target)}$  scores are listed in the result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore the usual interpretation of z-scores is as follows:

| z | < 1good</li>
1 < | z | < 2satisfactory</li>
2 < | z | < 3questionable</li>
3 < | z | unsatisfactory</li>

### 4 EVALUATION

No problems were encountered during the execution of this proficiency test. Three laboratories reported the test results after the final reporting date. One laboratory reported no test results. And not all laboratories were able to perform all analyses requested. Finally, 18 laboratories did report 222 numerical test results. Observed were 7 outlying test results, which is 3.2%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

## 4.1 EVALUATION PER TEST

In this section the results are discussed per test. For the Density determination a not normal distribution was found. For many other tests the number of reported test results was too small to determine whether the data set was normally distributed.

- <u>Aromatics</u>: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of D6379:11.
- <u>Colour</u>: Five laboratories reported a test result of "Blue". No further conclusions were drawn.

Copper Corrosion: No conclusions were drawn. All participants agreed on result 1.

- <u>Density @15°C</u>: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of D4052:11 and with the much more strict requirements of the previous version D4052:02e1.
- <u>Distillation:</u> This determination is not problematic. No statistical outliers were observed and all calculated reproducibilities, except for 10% evaporated, are in good agreement with the requirements of D86:11a.
- Existent Gum: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility, after rejection of the statistical outlier, is in good agreement with the requirements of ASTM D381:09.
- <u>Freezing Point:</u> In this determination no problems have been observed. All reporting participants agreed on a result below -58°C. This value is the specification for freezing point according to the test scope D910:11.

- <u>Heat of Combustion:</u> This determination was very problematic. One statistical outlier was observed and one result was excluded for statistical evaluation as the test method used ASTM D4529 is not comparable with ASTM D3338. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of D3338:09. The small number of test results may (partly) explain the large spread.
- Lead as Pb: This determination was problematic for two laboratories. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of D3341:05.
- Lead as Tel: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of D3341:05. IIS converted Lead as Pb test results to Lead as Tel test results for laboratories with codes 120, 496, 1016 and 1521, in order to create a more significant evaluation.
- Lead precipitate: No significant conclusions were drawn. The four reporting laboratories agreed on a value "less 1 mg/100ml".
- <u>MON:</u> This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of D2700:10.
- <u>Potential Gum</u>: No significant conclusions were drawn. One statistical outlier was observed. The other seven laboratories agreed on a value "less 1 mg/100ml".
- <u>Sulphur</u>: This determination may be problematic for two laboratories. Only two laboratories reported a numerical result between 0.036 %M/M and 0.19 %M/M. However, nine other laboratories reported that no sulphur was detectable: <0.0005 %M/M.
- <u>Water reaction:</u> This determination may be not problematic. All participants reported 1 or below 1. Actually, the rating numbers 1, 1b, 2, 3 and 4 should be used for appearance according to D1094:07.

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average values, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards) are compared in the next table.

Parameter	unit	n	mean	2.8 * sd	R (lit)
Aromatics	%V/V	6	13.4	2.2	1.6
Colour		5	blue	n.e	n.e
Copper Corrosion 2 hrs/100 °C		16	1	n.e	n.e
Density @15°C	kg/m <sup>3</sup>	16	710.9	0.4	3.3
Distillation @ 760 mm Hg	-				
- Initial Boiling Point	°C	17	36.7	3.8	5.2
- 10% evaporated	°C	17	63.8	3.9	3.2
- 40% evaporated	°C	17	97.2	2.0	unknown
- 50% evaporated	°C	17	103.8	1.5	1.9
- 90% evaporated	°C	17	125.9	2.0	3.1
- Final Boiling Point	°C	17	152.2	2.5	6.8
Existent Gum	mg/100ml	10	0.5	0.7	(2.2)
Freezing Point	°C	10	<-58	n.e	2.5
Heat of Combustion (Net)	MJ/kg	6	43.758	0.115	0.046
Lead as Pb	g/l	8	0.55	0.01	0.03
Lead as Tel	ml/l	8	0.52	0.01	0.03
Lead participate content	mg/100ml	4	<1	n.e	(1)
MON (lean mixture)		6	102.2	3.3	0.9
Potential Gum	mg/100ml	4	0.8	0.7	(3.0)
Sulphur	%M/M	9	< 0.0003	n.e.	n.e.
Water reaction interface	ml	9	n.e	n.e	n.e

table 3: performance evaluation sample #12050

\*() = near or below the detection limit

Without further statistical calculations, it can be concluded that for several tests there is not a good compliance of the group of participants with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2012 WITH PREVIOUS PT

	April 2012	May 2011
Number of reporting labs	18	13
Number of results reported	222	163
Statistical outliers	7	2
Percentage outliers	3.2%	1.2%

table 4: Comparison with previous proficiency tests

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Parameter	April 2012	May 2011		
Aromatics		n.e.		
Density @15°C	++	++		
Distillation @ 760 mm Hg	+	++		
Existent Gum	(++)	++		
Heat of Combustion (Net)				
Lead as Pb	++			
Lead as Tel	++			
MON (lean mixture)		n.e.		
Potential Gum	(++)	n.e.		
Sulphur	n.e.			

table 5: comparison determinations against the standard requirements

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- -- : group performed much worse than the standard
- n.e.: not evaluated

### **APPENDIX 1**

Determination of Aromatics on sample #12050; results in %V/V

lab	method	value	mark	z(targ)	Ì
120	moniou		mant	_(targ)	
225					
273					
340	D6379	12.3		-1 99	
353	20010				
445					
447	D1319	13.0		-0.75	
463	D1319	13.0		-0.75	
496					
606					
631					
823					
962					
1016					
1017					
1080	in house	14.23		1.45	
1293					
1521	EN15553	13.88		0.82	
1833	D2854	14.1		1.21	
	normality	OK			
	n	6			
	outliers	0			
	mean (n)	13.42			
	st.dev. (n)	0.766			
	R(calc.)	2.15			
	R(D6379:11)	1.57			



# Determination of Colour on sample #12050

lab	method	value	mark	z(targ)	remarks
120	Visual	blue			
225					
273					
340					
353					
445	Visual	blue			
447					
463					
496					
606					
631					
823	Baaaa				
962	D2392	blue			
1016					
1017	D4500				
1080	D1500	0.0			
1293					
1521	IP17	3.1 Diue			
1033	D2392	blue			
	normality	ne			
	normanty	n.e.			
	outliers	ne			
	mean (n)	hlue			
	st dev (n)	n e			
	R(calc)	n.e.			
	R(D2392.11)	n e			
	N(B2002.11)				

# Determination of Copper Corrosion on sample #12050

lab	method	value	mark	z(targ)	remarks	
120	D130	1a				
225	D130	1a				
273	D130	1a				
340	D130	1a				
353	D130	1a				
445	D130	1a				
447	D130	1a				
463	D130	1a				
496	D130	1a				
606						
631	D130	1a				
823	D130	1a				
962	D130	1a				
1016	D130	1a				
1017						
1080	D130	1b				
1293						
1521	D130	1				
1833	D130	1a				
	normality	n.a.				
	n	16				
	outliers	n.a.				
	mean (n)	1 (1a)				
	st.dev. (n)	n.a.				
	R(calc.)	n.a.				
	R(D130:10)	n.a.				

# Determination of Density @ 15°C on sample #12050; results in kg/m<sup>3</sup>

lah	method	value	mark	z(targ)	remarks								
120	D4052	710.9		0.01									
225	D4052	710.0		0.01									
220	D4052	710.7		-0.17									
273	D4052	711.1		0.18									
340	D4052	710.91		0.01									
353	D4052	710.8		-0.08									
445	D4052	710.8		-0.08									
447	D4052	710.8		-0.08									
463	D4052	711.10		0.18									
496	D4052	710.89		0.10									
430	D4052	710.03	C(0.04)	0.00									
000	D4052	710.3	G(0.01)	-0.51									
631													
823	D4052	711.0		0.09									
962	D4052	711.1		0.18									
1016	D4052	710.9		0.01									
1017													
1080	D4052	710.8		-0.08									
1293	ISO12185	710.89		0.00									
1521	D4052	710.00		0.00									
1021	D4052	710.0		-0.08									
1833	D4052	710.8		-0.08									
		_											
	normality	OK											
	n	16											
	outliers	1											
	mean (n)	710 80											
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	R(D4052:11)	3.25			Compare	K(D405)	2:02e1)	= 0.50					
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	- ASTM-D4052:11												
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713 -													
712													
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708 +													
707 -													
706 -													
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## Distillation @ 760 mm Hg (automated + manual) on sample #12050.

lab	method	IBP	mark	1 <b>0</b> %	mark	40%	mark	<b>50%</b>	mark	90%	mark	FBP	mark
120	D86-A	35.31		63.08		97.03		103.61		125.36		153.50	
225	D86-M	39.0		62.0		96.0		103.0		125.0		151.0	
273	D86-A	36.1		64.1		96.6		103.2		125.7		152.3	
340	D86-A	36.5		64.5		97.9		104.2		126.6		152.8	
353	IP123-A	35.4		62.1		96.8		103.4		126.1		152.7	
445	D86-A	38.1		62.7		96.9		103.7		125.9	С	152.7	
447	D86-A	35.2		62.4		97.5		104.0		126.1		151.6	
463	D86-A	36.9		66.7		97.1		104.3		126.1		151.6	
496	D86-A	38.8		65.8		98.7		105.0		127.8		152.6	
606	D86-A	34.9		63.2		98.0		104.3		126.7		153.3	
631	D86-M	38.0		64.0		97.0		104.0		126.0		153	С
823	D86-A	38.1		63.3		97.8		103.9		125.2		150.2	
962	D86-M	36.0		64.0		97.0		103.5		125.0		153.0	
1016	D86-A	35.9		64.1		97.3		103.8		125.6		151.3	
1017													
1080	D86-A	36.7		62.3		96.3		103.2		125.2		151.3	
1293													
1521	D86-A	35.3		64.1		97.8		104.1		126.2		152.3	
1833	D86-	37.9		66.1		96.4		103.0		125.4		152.5	
	normality	OK		OK		OK		OK		OK		OK	
	n	17		17		17		17		17		17	
	outliers	0		0		0		0		0		0	
	mean (n)	36.71		63.79		97.18		103.78		125.88		152.22	
	st.dev. (n)	1.355		1.397		0.699		0.534		0.717		0.904	
	R(calc.)	3.79		3.91		1.96		1.50		2.01		2.53	
	R(D86:11a)	5.23		3.20		unknown		1.88		3.11		6.78	

Lab 445 first reported 115.7

Lab 631 first reported 157.0

Z-scores						
lab	IBP	10%	40%	50%	90%	FBP
120	-0.75	-0.62		-0.25	-0.47	0.53
225	1.22	-1.57		-1.16	-0.79	-0.50
273	-0.33	0.27		-0.86	-0.16	0.03
340	-0.11	0.62		0.63	0.65	0.24
353	-0.70	-1.48		-0.56	0.20	0.20
445	0.74	-0.96		-0.11	0.02	0.20
447	-0.81	-1.22		0.33	0.20	-0.26
463	0.10	2.54		0.78	0.20	-0.26
496	1.12	1.76		1.82	1.73	0.16
606	-0.97	-0.52		0.78	0.74	0.45
631	0.69	0.18		0.33	0.11	0.32
823	0.74	-0.43		0.18	-0.61	-0.83
962	-0.38	0.18		-0.41	-0.79	0.32
1016	-0.43	0.27		0.03	-0.25	-0.38
1017						
1080	-0.01	-1.31		-0.86	-0.61	-0.38
1293						
1521	-0.76	0.27		0.48	0.29	0.03
1833	0.64	2.02		-1.16	-0.43	0.12



# Determination of Existent Gum on sample #12050; results in mg/100ml

lah	mothod	voluo	mark	=(torg)	romorko	
	Door	value	mark	Z(larg)	rendrks	
120	D381	0.53		0.05		
225	D381	2.0	G(0.01)	1.96		
273						
340	D381	0.4		-0.12		
353	D381	<1				
445	D381	0.9		0.53		
447	D381	1		0.66		
463	D381	0.4		-0.12		
496	D381	0.3		-0.25		
606	D381	0.2		-0.38		
631	D381	0.4		-0.12		
823	D381	<1.0				
962	D381	0.40		-0.12		
1016	D381	<1				
1017						
1080	ISO6246	<1				
1293						
1521	D381	<1				
1833	D381	0.4		-0.12		
	normality	not OK				
	n	10				
	outliers	1				
	mean (n)	0.49				
	st dev (n)	0.256				
	R(calc.)	0.200				
	R(D381.09)	2 15				
		2.10				





# Determination of Freezing Point on sample #12050; results in °C

lab	method	value	mark	z(targ)	remarks		
120	D2386	<-75					
225	D2386	<-70					
273	22000						
340	D2386	<-70					
353							
445							
447	D2386	<-58					
463	D2386	<-65.0					
496	D2386	<-77.5					
606							
631	D2386	<-58.0					
823	D2386	<-60.0					
962							
1016	D2386	<-58.0					
1017							
1080							
1293							
1521	D2386	<-60					
1833							
	normality	n.a.					
	n	10					
	outliers	n.a.					
	mean (n)	<-58					
	st.dev. (n)	n.a.					
	R(calc.)	n.a.					
	R(D2386:06)	2.5					

## Determination of Heat of Combustion (Net) on sample #12050; results in MJ/kg

		_	-		
lab	method	value	mark	z(targ)	remarks
120	D3338	43.737		-1.29	
225	D3338	42.79	G(0.01)	-58.93	
273					
340	D3338	43.8012		2.62	
353					
445					
447	D3338	43.769		0.66	
463	D3338	43.76		0.11	
496					
606					
631	D3338	43.7925		2.09	
823	D3338	43.689		-4.21	
962					
1016					
1017					
1080					
1293					
1521	D4529	43.908	ex	9.12	Result excluded as test method is not comparable with D3338
1833					
	normality	OK			
	n	6			
	outliers	1			
	mean (n)	43.7581			
	st.dev. (n)	0.04095			
	R(calc.)	0.1147			
	R(D3338:09)	0.0460			



## Determination of Lead as Pb on sample #12050; results in g/l

lab	method	value	mark	z(targ)	remarks
120	D5059	0.55		0.40	
225					
273	D3341	0.54		-0.59	
340					
353	IP270	0.5478		0.18	
445	IP270	0.5389		-0.70	
447	IP362	0.50	G(0.01)	-4.56	
463	D3341	0.5472		0.12	
496	D3341	0.55315		0.71	
606					
631					
823					
962					
1016	D5059	0.544		-0.20	
1017					
1080					
1293					
1521	D5059	0.5468		0.08	
1833	EN237	41.8	G(0.01)	4090.13	Probably reported in a different unit?
		<u></u>			
	normality	OK			
	n	8			
	outliers	2			
	mean (n)	0.5460			
	st.dev. (n)	0.00482			
	R(calc.)	0.0135			
	R(D3341:05)	0.0282			





## Determination of Lead as TEL on sample #12050; results in ml/l

lab	method	value	mark	z(targ)	remarks
120		0.52		0.36	converted by iis from the reported Lead as Pb result
225					
273	D3341	0.511		-0.56	
340					
353	IP270	0.5182		0.18	
445	IP270	0.5098		-0.68	
447					
463	D3341	0.5176		0.11	
496		0.5233		0.70	converted by its from the reported Lead as Pb result
606					
631					
823					
962					an example differ the form the memory of the sector <b>D</b> is sector.
1016		0.515		-0.15	converted by lis from the reported Lead as Pb result
1017					
1080					
1293		0.517		0.05	converted by its from the reported Load on Dh repult
1021		0.517		0.05	convened by its from the reported Lead as PD result
1055					
	normality	ОК			
	n	8			
	outliers	0			
	mean (n)	0.5165			
	st.dev. (n)	0.00448			
	R(calc.)	0.0125			
	R(D3341:05)	0.0274			
0.56 -					





# Determination of Lead precipitate content on sample #12050; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
120					
225					
273					
340					
353					
445					
447	D873	<1			
463					
496					
606					
631					
823	D873	0.3			
962	D873	<1			
1016					
1017					
1080					
1293					
1521	D873	<1			
1833					
	normality	n.e.			
	n	4			
	outliers	n.e.			
	mean (n)	<1			
	st.dev. (n)	n.e.			
	R(calc.)	n.e.			
	R(D873:02)	(1)			

### Determination of MON (lean mixture) on sample #12050

lab	method	value	mark	z(targ)	remarks
120					
225					
273					
340	D2700	101.5		-2.26	
353					
445	D2700	100.6		-5.06	
447	D2700	102.8		1.79	
463	D2700	101.9		-1.01	
496	D2700	102.55		1.01	
606					
631					
823					
962					
1016					
1017					
1080					
1293					
1521	D2700	104.0		5.52	
1833	D2700	128.0	G(0.01)	80.19	
	normality n outliers mean (n) st.dev. (n) R(calc.) R(D2700:10)	OK 6 1 102.2 1.17 3.3 0.9			



2 1.5 1

0.5

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631

823

# Determination of Potential Gum on sample #12050; results in mg/100ml

lab	method	value	mark	z(targ)	remarks
120					
225					
273					
340	D873	3.0	G(0.05)		
353					
445					
447	D873	1			
463	D873	<1			
496					
606					
631	D873	0.4			
823	D873	0.8			
962	D873	0.80			
1016	D873	<1			
1017					
1080					
1293					
1521	D873	<1			
1833					
	normality	unknown			
	n	4			
	outliers	1			
	mean (n)	0.75			
	st.dev. (n)	0.252			
	R(calc.)	0.71			
	R(D873:02)	(3.00)			
	(	( )			
3.5 T					
3 -					x
2.5 -					

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447

340

# Determination of Sulphur content on sample #12050; results in %M/M

		-		-
lab	method	value	mark z(targ	remarks
120	D7039	0.0002		-
225				
273				
340	D2622	<0.0001		
353				
445	D5453	<0.0001		
447	IP490	0.00005		
463	D4294	0.0586		- False positive result?
496	D2622	<0.0005		•
606				
631				
823				
962	D2622	0.0516		- False positive result?
1016	D2622	< 0.0003		
1017				
1080	D5453	<0.0001		
1293				
1521	D2622	< 0.0003		
1833	D5453	0.000012		
	normality	not OK		
	n	9		
	outliers	0		
	mean (n)	<0.0003		
	st.dev. (n)	n.a.		
	R(calc.)	n.a.		
	R(D2622:10)	n.a.		Application range >0.0003 %M/M

# Determination of Water reaction interface on sample #12050; results in ml

lab	method	value	mark	z(targ)	remarks
120	D1094	<0.5			
225					
273	D1094	0			
340	D1094	0.0			
353					
445	D1094	1			
447	D1094	<0.5			
463	D1094	1.0			
496					
606					
631	D1094	0.5			
823	D1094	<0.5			
962					
1016					
1017					
1080					
1293					
1521	D1094	0.5			
1833					
	normality	n.a.			
	n	9			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D1094:07)	n.a.			

### **APPENDIX 2**

### Number of participants per country

- 1 lab in BELGIUM
- 1 lab in CÔTE D'IVOIRE
- 1 lab in FRANCE
- 1 lab in GERMANY
- 1 lab in HUNGARY
- 1 lab in IRELAND
- 1 lab in KOREA
- 1 lab in MALAYSIA
- 1 lab in PHILIPPINES
- 1 lab in POLAND
- 1 lab in SAUDI ARABIA
- 1 lab in SOUTH AFRICA
- 1 lab in SWEDEN
- 1 lab in THE NETHERLANDS
- 2 labs in TURKEY
- 1 lab in U.S.A.
- 2 labs in UNITED KINGDOM

### **APPENDIX 3**

### Abbreviations:

С	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
ex	= excluded from calculations
Е	= error in calculations
n.e.	= not evaluated
W	= withdrawn
fr.	= first reported
U	= reported in different unit
SDS	= Safety Data Sheet

## Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
- 2 ASTM E178-02
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- 4 ISO 5725-86
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- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 12 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No4 January 2001
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)
- 15 H. Verplaetse and M. Lacourt, Accred Qual Assur (2006) 11:521-522